REQ. NO. 2-9019

XRAL

CONTRACT REPORT NO. OSQ 82-00020

METHODS OF ANALYSIS OF REFERENCE MATERIALS UTS-1 TO UTS-4

- General comments: All analyses were performed in one group. Other Canmet and international reference materials (RM) were run along with the samples. In some cases they were used as controls. In some other cases the whole calibration is based on a large number of these RM.

- Fe, Ti, Al, Ca determinations

  1.3 grams of sample are mixed with 5 grams of Li_2BO_4, fused into 40 mm glass button. This button is analyzed on a Philips PW1600 simultaneous x-ray fluorescence spectrometer calibrated with approximately 50 RMs. Matrix correction based on the alpha coefficients.

- Ba, S determination

  8 grams of sample and 2 binder pellets are mixed and pressed into a 32 mm pellet. A Philips PW1410 sequential x-ray fluorescence spectrometer calibrated with several international RM is used in this case. Ba is done using W excitation and S is done using Cr excitation.

- U determination

  1 gram sample is irradiated at McMaster University reactor and determination done by NAS using delayed neutron counting. Canmet RM are run regularly to check the calibration.

- Th, As determination

  0.5 gram sample is analyzed by instrumental neutron activation analysis. Several RM are used to check the calibration.

- Ni determination

  0.25 gram sample are digested using H_2SO_4 - HNO_3 - HF taking it to fumes, then add more HNO_3 and HF, taken to dryness, residue taken up with HNO_3 - HCl.
  The solutions were run on atomic absorption spectrometer (Perkin-Elmer 4000). Control samples are digested and analysed in same batch.

- SO_4 determination

  1 gr sample is digested using HCl, followed by addition of $BaCl_2$. Gravimetric determination of $BaSO_4$.

URANIUM TAILINGS UTS-1 Bottle # 1431

Constituent	Sample Size (g)	Test portion 1	2	3	4	5
Fe (total) %	1.3	4.98, 5.01	5.01, 4.96	4.94, 4.97	4.94, 4.93	4.95, 4.98
Ti %	1.3	0.53, 0.52	0.54, 0.53	0.52, 0.53	0.52, 0.52	0.53, 0.54
Al %	1.3	6.24, 6.26	6.27, 6.25	6.23, 6.25	6.22, 6.20	6.24, 6.27
Ca %	1.3	5.22, 5.23	5.25, 5.22	5.21, 5.20	5.18, 5.20	5.23, 5.25
Ba ppm	8	360, 340	340, 360	340, 380	360, 340	340, 360
U ppm	1	51.1, 52.4	52.2 , 48.1	51.5 47.0	50.1, 52.9	48.9, 52.5
Th ppm	0.5	142, 128	126, 135	133, 142	140, 138	138, 138
S (total) %	4	1.67, 1.58	1.64, 1.65	1.68, 1.61	1.66, 1.70	1.58, 1.65
SO_4^{2-} %	1	2.59, 2.63	2.52, 2.52	2.40, 2.36	2.62, 2.65	2.57, 2.57

URANIUM TAILINGS UTS-2 Bottle # 858

Constituent	Sample Size (g)	Test portion 1	2	3	4	5
Fe (total) %	1.3	3.05, 3.06	3.08, 3.12	3.08, 3.09	3.05, 3.10	3.06, 3.02
Ti %	1.3	0.22, 0.22	0.22, 0.22	0.22, 0.21	0.22, 0.22	0.22, 0.22
Al %	1.3	2.71, 2.69	2.69, 2.69	2.70, 2.67	2.68, 2.68	2.71, 2.72
Ca %	1.3	0.44, 0.44	0.44, 0.44	0.44, 0.44	0.44, 0.44	0.44, 0.44
Ba ppm	8	520, 520	520, 540	540, 520	520, 520	520, 500
U ppm	1	55.8, 56.2	57.8, 56.0	54.5, 56.2	55.7 57.2	57.8 55.4
Th ppm	0.5	160, 171	158, 170	172, 169	165, 162	166, 165
S (total) %	4	3.09, 3.03	3.11, 3.12	3.06, 3.07	3.13, 3.06	3.05, 3.10
SO_4^{2-} %	1	0.73, 0.66	0.69; 0.66	0.66, 0.69	0.72, 0.63	0.69, 0.61

URANIUM TAILINGS UTS-3 Bottle # 423

Constituent	Sample Size (g)	Test portion 1	2	3	4	5
Fe (total) %	1.3	3.43, 3.42	3.44, 3.37	3.38, 3.38	3.38, 3.33	3.40, 3.38
Ti %	1.3	0.24, 0.25	0.25, 0.24	0.24, 0.25	0.25, 0.24	0.25, 0.25
Al %	1.3	5.87, 5.87	5.90, 5.87	5.87, 5.88	5.87, 5.88	5.93, 5.90
Ca %	1.3	4.04, 4.07	4.04, 4.05	4.05, 4.05	4.05, 4.06	4.11, 4.03
Ba ppm	8	180, 200	180, 180	180, 200	180, 180	180, 180
U ppm	1	556, 546	553, 555	550, 551	548, 554	539, 560
Th ppm	0.5	9.2, 8.9	8.9, 8.9	9.8, 8.9	9.0, 8.9	8.9, 9.0
S (total) %	4	0.21, 0.23	0.21, 0.21	0.20, 0.23	0.19, 0.20	0.20, 0.21
SO_4^{2-} %	1	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01

URANIUM TAILINGS UTS-4 Bottle # 330

Constituent	Sample Size (g)	Test portion 1	2	3	4	5
Fe (total) %	1.3	2.75, 2.76	2.76, 2.77	2.73, 2.78	2.72, 2.74	2.73, 2.73
Ti %	1.3	0.26, 0.26	0.26, 0.27	0.26, 0.27	0.26, 0.26	0.26, 0.26
Al %	1.3	6.20, 6.20	6.21, 6.23	6.20, 6.23	6.20, 6.19	6.22, 6.21
Ca %	1.3	1.80, 1.82	1.81, 1.80	1.79, 1.82	1.79, 1.81	1.80, 1.80
Ba ppm	4	80, 80	100, 100	80, 100	80, 100	80, 100
U ppm	1	1039, NSS	1037, NSS	1028, NSS	1036, NSS	1035, NSS
Th ppm	0.5	14.6, 14.4	14.8, 14.2	14.9, 13.7	15.5, 13.7	15.2, 13.6
S (total) %	4	3.09 3.15	3.19, 3.19	3.14, 3.12	3.14, 3.07	3.08, 3.16
SO_4^{2-} %	1	5.30 5.26	5.20, NSS	5.40, NSS	5.20, NSS	5.31, 5.24
Ni ppm	0.25	130, 130	130, 130	130, 130	130, 130	130, 130
As ppm	0.5	41, 40	41, 41	44, 40	39, 42	39, 43